

**CALIFORNIA ENVIRONMENTAL PROTECTION AGENCY  
DEPARTMENT OF TOXIC SUBSTANCES CONTROL**

**HAZARDOUS WASTE TECHNOLOGY CERTIFICATION PROGRAM  
EVALUATION REPORT**

**File Contains Data for**  
**Site Characterization and Analysis Penetrometer System Thermal Desorption Sampler**  
**(SCAPS TDS)**  
**as an in-situ field screening technology**  
**PostScript Printers Only**  
**for the detection of selected volatile organic compounds**

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## Acronyms

CI	chemical ionization
CRREL	Cold Regions Research and Engineering Laboratory
DCE	dichloroethene
DSITMS	direct sampling ion-trap mass spectrometer
DTSC	California Department of Toxic Substances Control
EI	electron impact
ERDC-ECB	U.S. Army Engineer Research and Development Center, Environmental Chemistry Branch
ERDC-WES	U.S. Army Engineer Research and Development Center, Waterways Experiment Station
GC/MS	Gas Chromatograph/Mass Spectrometer
GC/PID	Gas Chromatograph/photoionization Detector
ITMS	ion-trap mass spectrometer
MDL	minimum detection limit
OPPTD	Office of Pollution Prevention and Technology Development
ORNL	Oak Ridge National Laboratory
PCE	perchloroethylene (tetrachloroethene)
PFTBA	perfluorotributylamine
PV	probe verification
QA/QC	quality assurance/quality control
QC	quality control
SCAPS	Site Characterization and Analysis Penetrometer System
SOP	standard operating procedure
TCE	trichloroethene (trichloroethylene)
TDS	thermal desorption sampler
VOCs	volatile organic compounds

## **1. FORWARD**

Chapter 412, Statutes of 1993, Section 25200.1.5., Health and Safety Code, enacted by Assembly Bill 2060, authorizes the California Department of Toxic Substances Control (DTSC) to certify the performance of hazardous waste environmental technologies. The purpose of the certification program is to provide an independent technical evaluation of technologies to identify those meeting applicable quality standards, so as to facilitate regulatory and end-user acceptance and to promote and foster growth of California's environmental technology industry. As part of this program, DTSC has evaluated a cone-penetrometer-based thermal desorption system for the detection of volatile organic compounds in soil. This report was prepared to provide the results of this evaluation. The evaluation is based on a detailed review of the technical documents and validation data submitted by the developer, and the validation data generated in field demonstrations in which DTSC participated.

## **2. INTRODUCTION**

The Site Characterization and Analysis Penetrometer System (SCAPS) Thermal Desorption Sampler (TDS) is a near real-time in-situ subsurface screening method for volatile organic compounds (VOCs). The technology was developed through the Tri-Service SCAPS program and is one of a planned family of sensors collectively called the Site Characterization and Analysis Penetrometer System, or SCAPS, that will combine remote sensors with a cone penetrometer platform to provide rapid, in-situ, subsurface measurements of many different contaminants. The method that was evaluated uses a Thermal Desorption Probe deployed with the cone penetrometer to thermally desorb VOCs from the soil. The thermal desorption probe is advanced with the tip of the cone penetrometer to sample successive discrete volumes of soil in-situ. Using helium gas, desorbed VOCs are transferred to the surface and concentrated onto a sorbent trap. Analysis for target analytes is performed on-board using an ion-trap mass spectrometer (ITMS). Spent soil samples are ejected before advancing the probe to each successive sampling depth.

The conventional or traditional approach to site characterization, which depends on collection of discrete soil and water samples followed by laboratory analyses, is usually a slow, iterative, and costly process. Significant delays occur in site characterization while samples are analyzed. Subsequent sample borings are completed with no knowledge of the results from other boring locations, or the process must stop to await results from previous sampling. The SCAPS TDS technology was designed to improve upon conventional site characterization by providing rapid qualitative to semi-quantitative information about the subsurface distribution of volatile organic contamination.

The Thermal Desorption Sampler technology was developed by the U.S. Army Engineer Research and Development Center, Waterways Experiment Station. The U.S. Department of Energy Oak Ridge National Laboratory developed the ion-trap mass spectrometer for application with the SCAPS TDS. Modifications to improve the SCAPS TDS system were made during the course of field studies that were considered in the certification evaluation. This evaluation report focuses on the equipment as it is presently configured and operated.

### **3. DESCRIPTION OF THE TECHNOLOGY**

The SCAPS TDS technology was developed for deployment with a standard cone penetrometer to provide near real-time semi-quantitative field screening analyses of volatile organic compounds (VOCs) in the vadose and capillary zones. In operation, the SCAPS TDS probe is pushed to a desired depth, an interior rod retracts the penetrometer tip, and a known volume of soil is collected in the sample chamber. The sample chamber is heated in-situ and purged with helium carrier gas to desorb the VOCs. The desorbed VOCs are transferred to the surface through an umbilical cable threaded through the cone penetrometer push rods. The VOCs are concentrated onto a sorbent trap for subsequent analysis by an ITMS on board the SCAPS truck. After desorption, the soil sample is expelled and the cone penetrometer is pushed to a new depth to where the sampling process is repeated. The SCAPS TDS technology consists of two primary components: (1) the Thermal Desorption Probe and associated sample collection equipment deployed with a cone penetrometer, and (2) an on-board ITMS and associated equipment for near real-time analysis of the samples collected on a sorbent traps. A detailed description of the system components follows.

#### **3.1 Support Platform and Cone Penetrometer (CP)**

The cone penetrometer (CP) platform is used to deploy the Thermal Desorption Sampler and houses all associated sample collection and analytical instruments. Typically, the CP is housed in a 20-ton truck; 20 tons is the static reaction force that the weight of the truck can exert on the pushrod of the CP. Within the truck, the CP and the on-board analytical system with computers are housed in separate compartments. The CP pushrod is composed of detachable 1-m long tubular sections which thread together one-by-one to lengthen the pushrod as it is advanced into the ground with a hydraulic ram against the weight of the truck. Typically, the CP sensors are advanced by attaching successive lengths of pushrod in 1-meter (m) lengths at a rate of up to 1 m per minute to a potential maximum depth (at present) of 50 m ( $\approx$  150 ft). Umbilical cable, containing the wires and tubes needed to control and operate the sensor, is threaded through the pushrods prior to deployment of the technology.

As the pushrod is withdrawn at the end of each push, the tube sections are steam-cleaned before being disconnected for storage. After all of the push rods are withdrawn from the ground, the push hole is grouted from the bottom up by lowering a 3/4-inch PVC tremie pipe to the bottom of the push hole and pouring a cement/bentonite slurry through the tremie pipe as it is being gradually withdrawn.

Other CP platform samplers used with the TDS technology but not included in this evaluation are direct push split sleeve samplers for soil verification sampling and soil stratigraphy sensors equipped with stress sensors. The industry standard of using the cone penetrometer to measure cone tip pressure and sleeve friction for determining soil characteristics and subsurface lithologic conditions is addressed in ASTM Method D-3441-86. For site characterization purposes, this method is generally used to identify optimal sampling depths and locations prior to use of the SCAPS TDS.

### **3.2 Thermal Desorption Sampler (TDS) Probe**

The TDS principle of operation involves thermally desorbing VOCs from a known soil volume in-situ. The probe design is a series of steel cylinders with gas channels and piston chambers made tight by o-rings. A schematic of the probe is given in Figure 1. A central actuator rod is held in place by locking lugs in the closed position while the probe is being pushed into the ground. Once the probe reaches sampling depth, the locking lugs are pneumatically released and the piston is retracted to open the sample chamber. At sampling depth the probe is pushed an additional 1.75-2 inches to sample a soil plug of known diameter and estimated volume. Depending upon soil density, the plug weight ranges from 3.5 to 5.0 g. A push of 1.9 inches results in a soil sample plug approximately 1 inch long in the sample chamber. The chamber is heated by a nichrome-wire-wrapped ceramic heater fitted with an inner stainless steel protective sleeve and a thermocouple to monitor temperature. The temperature of the ceramic heater can be controlled to stabilize and maintain specified soil temperatures in the sample chamber. Soil temperatures during sampling are maintained between 150 to 200°C, depending on the soil conditions and contaminants present at the site. Helium is introduced through stainless-steel tubing located along the inner wall of the outer housing at a rate of 50 ml/minute. The gas enters the sample chamber area from behind and below. It is preheated to temperatures between 150 and 200°C as it moves across the surface of the heater before sweeping upward over the soil plug to purge the VOCs as they are volatilized into the chamber. The gas carries the volatilized sample up through the analyte line and into the sample collection device at the surface. Once the soil has been desorbed, the plug is ejected by forcing a burst of high-pressure gas down the line while lowering the actuator rod. A sensor in the probe indicates the rod's position to the operator at the surface. After the spent soil is ejected and the actuator rod is locked in the closed position, the TDS is pushed to a new depth and the sampling process is repeated. Sampling times generally vary between 15 and 30 minutes depending on soil type and moisture content.

The TDS probe is linked to a manifold housed in the SCAPS truck at the surface by a 200-ft umbilical cable threaded through the 1-m sections of push rods. Gas flows, temperature, and position of the actuator rod are controlled and monitored at the surface through the manifold. Digital mass flow meters are used to control carrier gas flows into and from the probe sample chamber to prevent sample losses and to ensure that soil gas outside the sample chamber is not introduced into the sampling process. The volatilized sample is collected on a sorbent trap attached to a manifold inside the SCAPS truck prior to analysis by ITMS.

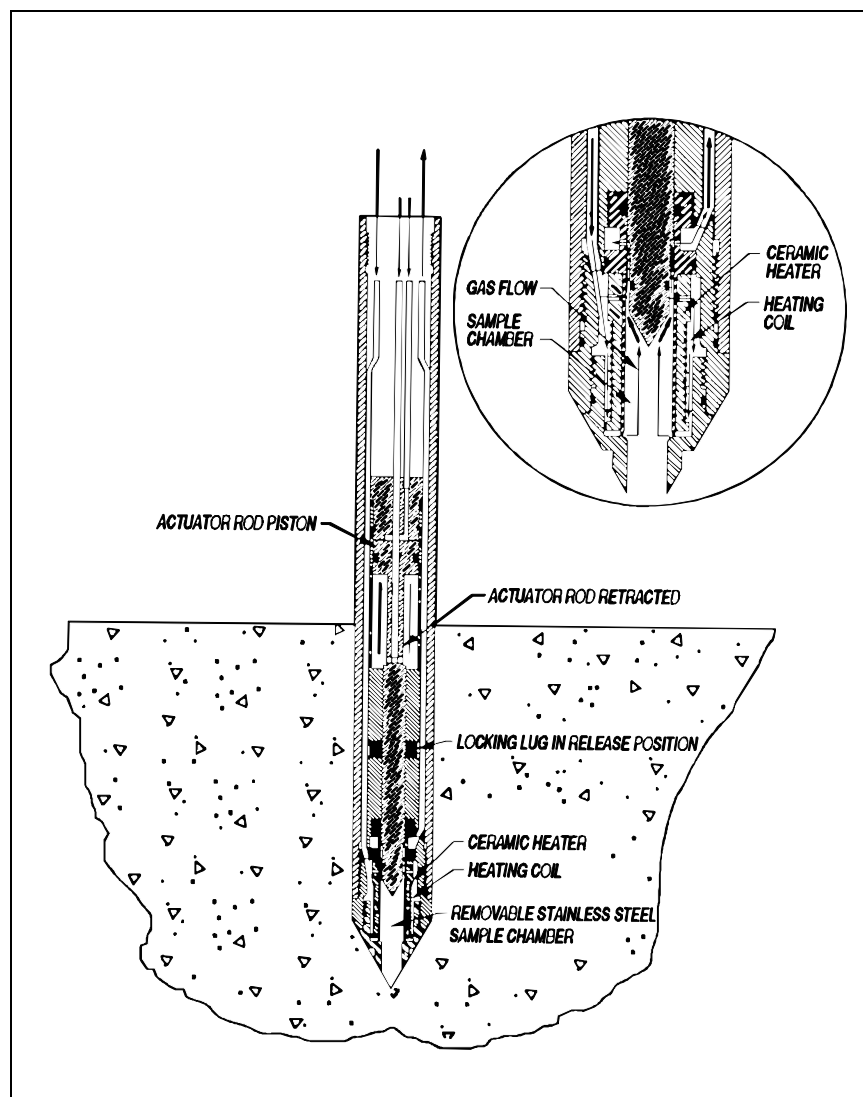
### **3.3 Ion-Trap Mass Spectrometer**

The on-board ITMS is comprised of a quadrupole ion-trap mass spectrometer, a capillary restrictor interface, and different types of sample inlets for use with gas (air or soil gas), soil, and water for on-site measurement and monitoring. Analysis of the TDS samples concentrated on sorbent traps is performed with the use of a Teledyne 3DQ or a Finnigan ITMS 40 operating in the electron impact (EI) mode or chemical ionization (CI) mode. An OI Analytical model 4560 purge-and-trap sample concentrator that has been modified for use with the TDS is used as the sample introduction device. Unlike most mass spectrometry systems, the analyte vapors purged from the sorbent trap are directly subjected to mass spectrometry without prior separation. Therefore, analyte identification is performed entirely by means of mass ion identification. Analytes with the same quantitation mass ions cannot be distinguished from each other and are reported as totals similarly to analytes co-eluting in GC techniques.



Details of the analytical method for in-situ measurement of VOCs in soil using SCAPS TDS and ITMS are described in the document, "Direct Sampling Ion-Trap Mass Spectrometry for the Measurement of Volatile Organic Compounds in Water, Soil and Air," Draft EPA SW-846 Method 8265, 1997<sup>(21)</sup>. This method was developed by Oak Ridge National Laboratory (ORNL) and field tested by the U.S. Army Corps of Engineers with a variety of direct sampling devices, including the SCAPS TDS. Based on this effort, U.S. EPA is now considering Method 8265-Volatiles by Direct Sampling Ion-Trap Mass Spectrometry (ITMS) for inclusion in the Update IV B of EPA Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846).

The draft Method 8265 identifies 30 target analytes and their corresponding quantitation ions that can be analyzed by ITMS. Tables 1 and 2 (Sections 5 and 6) indicate the analytes analyzed by the SCAPS TDS during past field studies.



**Figure 1.** Schematic of SCAPS TDS Probe

## 4. EVALUATION OF TECHNOLOGY: GENERAL CONSIDERATIONS

The technology evaluation considered performance parameters, including sensitivity, specificity, precision, accuracy, and reliability. A major part of the evaluation is the comparison of results obtained using the technology with those obtained using accepted reference methods. These issues affect all field validations and are therefore discussed before a review of the validation studies.

### 4.1 Scientific Principles of the Technology

The technology involves the application of accepted and well-documented scientific principles of thermal desorption, collection of VOCs with sorbent traps, and analysis of VOCs using mass spectrometry. Mass spectrometry in these validation studies was carried out either with a Teledyne 3DQ ITMS or with a Finnigan ITMS 40. These principles upon which the technology is based, are basically sound.

### 4.2 TDS Operation

The standard operating procedures for the in-situ measurement of VOCs in soil using SCAPS TDS have been described in US EPA SW-846 Draft Method 8265<sup>(3,19)</sup>, by Myers, et. al.<sup>(4)</sup>, and in the technology demonstration plans prepared for this project<sup>(10,12,13)</sup>. Currently recommended standard operating procedures based on past development work are described in the “Tri-Service Site Characterization and Analysis Penetrometer System (SCAPS) Validation of the Thermal Desorption Sampler for Volatile Organic Compounds, Instruction Report,” dated 1999<sup>(30)</sup>.

A routine sampling scheme consists of a TDS sample, followed by a 5 minute purge, and a 5 minute blank to monitor system carryover. The TDS probe soil sampling chamber is heated to 150°C prior to being pushed into the ground with the cone penetrometer. To collect a sample the TDS probe is advanced to the desired depth and the probe tip is retracted by means of an actuator rod and piston assembly. The exposed sample chamber is then pushed 1.8 inches into the formation to collect a sample. The temperature of the sample chamber is maintained between 150°C and 200°C for a period of 15 to 30 minutes depending on soil type and moisture content. Dry helium carrier gas flows through the sample chamber at 50 ml/minute to transfer the volatilized compounds onto a sorbent trap connected to a manifold in the SCAPS truck. The carrier gas flow rates are monitored and controlled to ensure that the flow out of the probe is within 5 ml/minute of that into and from the probe. At the end of sampling the sorbent trap is removed for subsequent analysis by ITMS. After each sampling event, the probe is raised approximately 1 foot using the cone penetrometer and a burst of high pressure gas is used to eject the spent soil plug and to help flush the sample chamber of any residual VOCs or moisture. Before proceeding to the next sampling depth the system is purged with dry helium for 5 minutes while maintaining the chamber temperature at 150°C. A 5 minute blank sample is collected if VOCs were present in the previous sample to confirm that no residual contaminants remain in the system before proceeding with the next sampling event.

#### 4.2.1 System Recovery Check

The standard operating procedure (SOP) requires that the TDS system recovery be checked daily before the probe is pushed into the ground. After initial warmup the TDS probe sample chamber is fitted with a gas-tight injection port. A blank sample is taken by trapping a 10-minute

helium purge. The TDS system was evaluated each day by injecting 250 ng gas mixture consisting of target analytes. Results from the daily system spikes were expressed in percentages. Gaseous standards are prepared on-site daily using Tedlar bags and neat standards. After the spike, the system is purged and a blank sample is collected.

#### *4.2.2 High concentration carryover*

High level subsurface VOC contamination may present a problem for normal continuous operation. Interference from a previous sampling or residual VOC analytes remaining in the transfer lines may cause cross contamination between successive runs. A contaminated transfer line can be cleaned by purging with helium or nitrogen or replaced with an extra umbilical cable. Therefore, blanks are analyzed after purging to ensure there are no residuals remaining between successive sampling events.

#### *4.2.3 Carrier Gas Flow Rate*

Helium is introduced into the probe sample chamber at a rate of 50 ml/minute. The flow rate of carrier gas from the sample chamber containing desorbed VOCs is maintained at an equal rate to ensure integrity of the sampling process (i.e. to maintain a closed system with no losses out of the system or introductions into the system). These two flow rates are maintained at the same flow rate using separate mass flow controllers. If the rates are not equal or balanced, a potential exists for sample losses or contamination from soil gas.

#### *4.2.4 Matrix Effects and Interferences*

##### *4.2.4.1 Moisture Content.*

During sampling, soil moisture is driven off as water vapor. This vapor has been observed condensing in the heated zone in the upper region of the TDS sample chamber. The burst of high-pressure gas used to help eject the spent soil plug flushes the lower analyte lines of any condensation and begins drying the air above the sample chamber. Generally, any moisture remaining in the sampling system is expelled during the heated purge step between samples. Soil moisture also effects the sampling time. Soils at or near the water table, or otherwise high-moisture soils, require longer heating times to reach and maintain the targeted sampling temperature. Traps for samples taken at or below the water table may need to be dried unheated with a stream of inert gas before being desorbed directly into the ITMS or eluted with methanol.

##### *4.2.4.2 Soil Type*

Thermal desorption of an analyte may be affected by variations in soil type as well as moisture content. Laboratory studies <sup>(2)</sup> indicate that the percent recovery with the TDS probe is somewhat reduced in clay soils versus sandy soils with the same moisture content. These results are based on the same sampling duration (30 minutes) and temperature (100 °C). It should be noted that these results were obtained before the system was field tested, after which it became apparent that temperatures higher than 150 °C were required to compensate for the cooling effect from the soil mass.

#### *4.2.5 Grouting of Push Hole*

Each SCAPS TDS push hole is immediately grouted upon removal of the push rod string to prevent contaminant migration along this potential pathway. A tremie pipe is used to grout the SCAPS TDS push hole from the bottom up, using a cement/bentonite slurry.

### **4.3 ITMS Operation**

Desorbed VOC samples are collected on OI Analytical style No. 9 traps filled with a mixture of Tenax, silica gel and charcoal. The trap is attached directly to the TDS manifold for sample collection. Samples are collected under a slight vacuum to balance the flow from the TDS Probe sample chamber with the inflow to probe sample chamber. Following sample collection, the OI trap is subject to VOC analysis using ITMS. The maximum capacity of the trap for contaminants has not been fully evaluated. However, it is important to note that the SCAPS TDS was intended to be used at lower contaminant concentrations, and there is a concern of breakthrough with the sorbent trap when the SCAPS TDS is to be used at soil contaminant concentrations around 100 mg/kg or greater.

#### *4.3.1 Instrument Calibration and Data Acquisition*

Detail of the procedures for the instrument calibration are given in Draft Method 8265<sup>(21)</sup>. For daily operation, the mass spectrometer needs to be checked and adjusted for the proper scan functions per instrument manufacturer's instructions. The basic ion scan function for generating alternative EI and CI mass spectra is listed in Table 1. (Ref. Table 4). The mass axis is calibrated with perfluorotributylamine (PFTBA) or other acceptable calibration compound set in full scan mode. The mass spectrum of air is evaluated for a system leak check. An instrument calibration file is acquired to ensure the proper setting of instrument tuning parameters. The critical operating parameters, such as sample ionization time, electron multiplier voltage (EM), and filament current, must be established to obtain the optimal sensitivity of the ITMS system response.

#### *4.3.2 Quantitative Calibration*

Detailed procedures for the quantitative calibration are given in Draft Method 8265<sup>(21)</sup>. The ITMS is calibrated by injecting volumes of mixed stock standard in various concentrations into a 5-ml aliquot of distilled water within the purge vessel of the OI Analytical purge-and-trap. The analytes are desorbed from the trap and a known mass of VOC analyte is transferred to the ITMS for quantification and calibration according to the established data acquisition program and data acquisition time. Calibration is performed daily using a set of calibration standards prepared from pure VOC compounds. Standard curves are developed from a blank and from up to five or six standard concentrations ranging from 25 to 1000 ng (only calibrated up to 750 ng in these studies), which are equivalent to 5-200 ng/g analyte in 5 g of soil. The calibration curve is generated by plotting the ITMS responses (ion abundance) of characteristic ions of each VOC analyte versus the concentration of the calibration standards. A linear fit is performed yielding slope, intercept, and correlation coefficient,  $R^2$ , which must be greater than 0.95. If the daily check standard is out of compliance, the system is checked for problems. The upper limit of the dynamic range for effective quantitation is 1000 ng/5 g of soil or 200 ppb)

Additionally, a single concentration calibration standard is run in triplicate at the beginning

of each day and again when any equipment is changed. If the standard deviation of the triplicate measurements exceeds 20 percent for replicate analyses of any single sample, that sample is rerun. If the deviation remains excessive, the system check standard is measured. If the check standard is out of compliance, system check out, debugging and recalibration are required.

#### 4.3.3 *Method Detection Limit and Sensitivity*

The calculation of Method Detection Limit (MDL) is described in EPA Draft Method 8265<sup>(21)</sup>. Noise, background and sensitivity are defined in terms of the parameters of the best fit regression line for the daily calibration data ( $y = mx + b$ ), as follows:

Sensitivity =  $m$ , the slope of the regression line

Background =  $b$ , the y-intercept of the regression line

Noise =  $s$ , the standard deviation of the fit of the regression line

The noise over the sensitivity ( $2 \times s/m$ ) is the detection level. For purposes of calculating the MDL, the in-situ soil sample wet weight is assumed to be 5 grams. With this approach the detection threshold of the system varies from site to site, but in the range of 0.1 to 0.3  $\mu\text{g/g}$  for the analytes investigated in field studies (see Table 6, Section 7).

As a field screening method, false negatives are a primary concern. To increase sensitivity and the confidence level near the detection threshold, and to minimize the probability of false negatives, low concentration standards are used to establish the linear regression.

#### 4.3.4 *Compound Identification and Quantitation*

Compound identification and quantitation are based on masses of selected characteristic ions for each target analyte. Since the analytes are not subjected to separation before entering the ion-trap mass analyzer, the mass spectrum of each compound is not available for compound identification. Quantitation of analytes is based on the integration over a fixed window of time (1.5 minutes) of the selected ion for a given analyte generated by either EI or CI. A list of characteristic ions of target compounds by ITMS is given in EPA Draft Method 8265<sup>(21)</sup>.

#### 4.3.5 *Sample Analysis*<sup>(4)</sup>

Collection and analysis of the TDS sample concentrated on the sorbent trap is routinely performed in one of two ways depending upon concentrations expected at the site. When low concentrations (below 0.5  $\mu\text{g/g}$ ) are expected, the sample is collected on an OI Analytical style No. 9 trap containing a mixture of Tenax, silica gel, and charcoal. For analysis, the trap is inserted into the purge-and-trap and desorbed directly into the ITMS. By design, the ITMS only uses approximately 4% of the gas flow entering the interface. The other 96% of the gas flow is vented through a port on the interface. When a second trap is attached to this port during sample desorption, the sample can be re-collected for reanalysis. Method detection limits for this technique are 10-20 ng/g of analyte on a mass of 5 g of soil. When concentrations are expected to be higher than 0.5  $\mu\text{g/g}$ , the TDS sample is collected on a Tenax trap and then eluted with 1 ml of purge-and-trap grade methanol. Aliquots of up to 0.1 ml are added to 5 ml of distilled water within the purge-and-trap

purge vessel and sparged into the ITMS for analysis. After methanol elution the trap is prepared for reuse by drying the Tenax with a stream of inert gas and heating for 10 minutes. The minimum method detection limits for this technique are 100-200 ng/g of analyte on a mass of 5 g of soil. Detection limits increase proportionally as the aliquot of methanol extract purged decreases in volume. The Tenax extraction technique is not suitable for the more volatile compounds such as trans-1,2-dichloroethene and volatile gases such as vinyl chloride. An OI Analytical No. 9 trap suitable for vinyl chloride is used in this case.

#### 4.3.6 Method precision and accuracy

Performance criteria and method recovery for ITMS are detailed in EPA draft method 8265<sup>(21)</sup>. Because of the heterogeneous distribution of contaminants in the subsurface, it is not possible to obtain precision data from in-situ sampling. The method precision was evaluated by analysis of spiked soils packed into a jig in the laboratory and by analysis of spiked standards injected directly into the heated sample chamber. These data were obtained using the Tenax traps and a gas chromatograph with a photo-ionization detector (GC/PID), and are presented in the following table<sup>(2)</sup>.

Comparison of recoveries from different soil types and two different moisture contents. Tubing was 1/16 inch stainless steel heated to 100 °C. Probe temperature was 100 °C with a flow rate of 40 mL/minute.

	Silt 10% M	Silt 20% M	Sand 10%M	Clay 10%M	Mean %Rec	8240 <sup>1</sup>
	% R (SE)	% R (SE)	% R (SE)	% R (SE)	% R (SD)	% R(SD)
Trans-1,2-dichloroethene	0.0 (0.0)	1.4 (0.2)	2.7 (0.6)	1.8 (0.6)	1.5 (1.1)	----
Benzene	96.5 (2.0)	69.3*(0.8)	92.6 (2.1)	87.8 (2.0)	86.6(12.1)	101 (7)
Trichloroethene	93.4 (1.2)	89.5 (2.5)	86.8 (1.6)	77.7 (7.3)	86.9 (6.7)	102 (7)
Toluene	102 (2.8)	94.1 (2.9)	95.9 (1.0)	88.3 (1.3)	95.0 (5.4)	102 (6)
Chlorobenzene	95.7 (1.0)	87.8 (3.2)	92.3 (0.1)	81.7*(0.3)	89.4 (6.1)	101 (4)
Ethylbenzene	92.9 (1.2)	93.9 (1.9)	91.2 (2.9)	81.7*(0.3)	89.9 (5.6)	----
m&p-Xylenes	101 (1.3)	90.5 (4.2)	96.8 (0.7)	90.0 (1.0)	94.6 (5.4)	----
o-Xylene	100 (1.3)	89.0*(1.0)	96.3 (0.6)	86.8*(0.5)	93.2 (6.4)	----
m&p- Dichlorobenzene	93.1 (2.1)	81.3 (3.8)	86.5 (0.7)	79.1 (2.9)	85.0 (6.2)	----
o-Dichlorobenzene	125 (8.2)	88.2 (1.8)	108 (10.3)	98.7 (7.4)	105 (15.6)	----

<sup>1</sup> Mean and standard deviation were calculated from matrix spike recoveries using EPA SW-846 Method 8240 by GC/MS.

\* Compounds exhibiting mean values dissimilar to the other treatments.

Notes: %R = percent recovery  
(SE) = standard error  
(SD) = standard deviation  
M = moisture content

## **4.4 System Limitations**

### *4.4.1 Saturated Zone*

Sampling below the water table may be problematic. It may take a long time to desorb samples in the saturated zone. In some saturated zone conditions (i.e., highly permeable zones) sampling times may be so long as to render the process impractical. Hydrostatic pressure will cause groundwater to replace initial water losses in the sample chamber due to volatilization. Potential also exists for flux of additional contaminants into the sample chamber from the contaminated ground water that enters into the sampling chamber. In such cases, results would be biased high.

### *4.4.2 Vertical Resolution*

Vertical sampling resolution is limited by the physical dimensions and operation of the TDS probe. After each sampling event the spent soil core is ejected from the sampler disturbing the soil below to some extent. Another concern is the heating of the soil in the sample chamber during the thermal desorption process and its affect on the surrounding soil. To avoid these concerns, a minimum vertical distance of 6 inches is maintained between sampling events.

### *4.4.3 Sampling Depth*

The sampling depth may be limited by lithologic conditions at a site. The maximum sampling depth of the SCAPS TDS is limited to 100 feet by the available length (200 ft.) of the umbilical which connects the TDS probe through the push rods to the sample collection manifold on the SCAPS truck.

### *4.4.4 Lithologic Conditions*

Use of the TDS probe is limited primarily to unconsolidated sediments where a cone penetrometer may be pushed to the desired sampling depths. Maximum depths for a cone penetrometer are typically not greater than 100 feet. Large cobbles embedded in finer grained sediments, hardpan layers, and other such lithologic features may further prevent penetration of the CP to desired sampling depths at a given site.

### *4.4.5 ITMS Operation*

Since the ITMS does not have a separation mechanism other than MS, compounds which produce identical primary characteristic ions or positional and geometric isomers, can not be identified by this system (e.g., 1,1-dichloroethene and 1,2-dichloroethene) unless a characteristic secondary ion is available for monitoring. Compounds with higher molecular weights may produce the same fragment ions which will increase the signal of target analytes. Therefore, this detection system can only be used to detect or to confirm the presence of target analytes but not for the identification.

## **4.5 Reliability**

Over the last several years, the SCAPS Thermal Desorption Sampler has been field tested at eight contaminated sites in different geographic locations with varied lithologic conditions. These field studies included 57 cone penetrometer pushes with the TDS probe to depths of 60 feet below ground surface to obtain over 160 discrete in-situ sample analyses. Over this period the system has been found to be reliable.

## **4.6 Reference Methods for Analysis of VOC-Contaminated Soils**

Conventional methods of subsurface characterization for VOC contamination involve obtaining discrete soil boring samples for off-site laboratory analysis of volatile organics by approved GC/MS or GC methods. Commonly, hollow-stem auger drilling techniques coupled with split-spoon core samplers are used to obtain discrete core samples with depth. In the past, 6-inch length core samples were capped, sealed and cooled to 4 °C for transport to an off-site lab for analysis. More recently methods have been approved for preserving soil samples with methanol in the field to prevent volatile losses which were known to occur with the previous method. For this study the verification samples consisted of subsamples of core samples that were preserved in the field with methanol using the method of Hewitt<sup>(27)</sup>. In this method approximately 4 ml of soil are immediately subsampled from the core into a preweighed 20 ml VOC vial that contains 5 ml of VOC grade methanol. The vial is sealed using a Teflon lined cap and the sample and vial are weighed to determine the soil sample weight. Once extracted into methanol and cooled to 4°C, these verification samples are shipped to an off-site laboratory for analysis by EPA Method 8260A. The laboratory should be certified by a state, federal or other recognized authority. EPA Method 5035<sup>(18)</sup> a closed-system purge-and-trap and extraction methodology for volatile organics is used in conjunction with Method 8260A analyses. It should be noted that the method for field preservation with methanol is essentially the first step of EPA Method 5035, except that it is performed in the field.

For the field verification studies, an ARA or Mostap direct push split spoon core sampler was used to obtain the verification core samples rather than use a conventional hollow-stem auger system. These samplers screw onto the end of the cone penetrometer push rod. To obtain the core samples separate pushes were completed adjacent to the SCAPS TDS push holes (horizontally offset by 8 to 12 inches). The core sample obtained with the Mostap sampler is 18 inches long and 1.8 inches in diameter.

## **4.7 Relation to Reference Methods for Analysis of VOC-Contaminated Soils**

The evaluation focused on comparing SCAPS TDS results with the results from soil core samples collected adjacent to the SCAPS CP push and analyzed by Method 8260A, the reference method. Both the ITMS and GCMS technologies involve the analysis of discrete samples and measure the amount of specific contaminants actually present in the subsurface. Each of the methods relies on similar assumptions, and compares what is measured to a similar standard, to provide an analysis of the true amount of contamination present in the subsurface.

The SCAPS TDS samples a discrete volume of soil (3 to 5 g sample), which is thermally desorbed in-situ. The thermally desorbed VOCs are then transferred through an analyte line using helium for collection onto a sorbent trap and subsequent analysis by the on-board ITMS.

The TDS probe sample chamber has an inner diameter of 0.438 inches and an effective length



of 0.986 inches. Due to potential physical and thermal disturbances to the surrounding soil when sampling, successive samples in a TDS push hole are taken at a minimum distance of 6 inches apart. Reference method samples are collected using standard core sampling technique. The split sleeve is taken from the core sampler, laid out on a flat surface adjacent to a ruler and samples are taken with a 10 mL syringe at preset distances from the top of the core. Samples are placed in preweighed vials containing 5 mL of methanol using procedures described by Hewitt<sup>(27)</sup>, and then transported to a laboratory for GCMS analysis. As with the TDS probe, there are limitations on the vertical resolution that can be achieved with successive core samples. These limitations are due to physical sampling disturbances caused by driving the split spoon sampler in the push hole (or boring if a hollow-stem auger is used), or due to poor sample retention with non-cohesive types of sediments. Because of these concerns the top and bottom portions of the core sample are not analyzed. In general, successive core samples for VOC analyses cannot be taken closer than a foot or more apart. Once retrieved, subsamples along the length of the core may yield better vertical resolution than the SCAPS TDS.

Fundamentally, there is little or no difference, if any, between what is actually measured by the SCAPS TDS and the reference method. The major difference between the two methods is the use of the ITMS versus the GCMS. Both are mass spectrometer methods. The GCMS includes a gas chromatography separation step which allows for positive identification of analytes. With the ITMS, analyte identification is performed entirely by means of mass ion identification. Compounds which produce identical primary characteristic ions or positional and geometric isomers, can not be positively identified.

The reference method is not able to analyze the same volume of soil as the in-situ SCAPS TDS discrete sample. The horizontal offset from the corresponding SCAPS TDS push hole adds an unknown level of uncertainty when comparing verification sample results with TDS sampling results. There is some uncertainty in determining the exact depth of a core sample due to potential errors in measurement, soil sloughing, and, near the groundwater table, heaving sands. There is an inherent difference in the soil sampled by TDS probe versus the soil core sample obtained for confirmation analysis. These differences can be significant, particularly where soil and contamination heterogeneities are present. Despite these limitations, the comparison of the reference method results to SCAPS TDS in-situ analyses was considered an acceptable method for assessing the technology's performance.

To assess whether subsurface heterogeneities exist at a sampling location, a subsample of each verification core sample was analyzed ex-situ using the SCAPS TDS. The subsample, termed the probe verification (PV) sample, was injected directly into the TDS probe sample chamber when the probe was above ground. This result was then compared to the in-situ TDS result to determine if a comparison of verification sample result with the in-situ TDS result was appropriate. Comparisons of results of the PV samples with the two adjacent methanol verification samples were also used to assess subsurface heterogeneity. Order of magnitude differences in between these results indicated a concern with heterogeneity at a sampling location.

#### **4.8 Evaluation of Accuracy**

Section 4.6 discusses the qualitative differences between the SCAPS TDS and reference methods. In general, the verification data is affected by a variety of uncertainties in physical parameters, such as the depth of physical sampling and horizontal offset of the TDS sample push hole from the verification sample push hole.

Vertical and horizontal variation in contaminant concentrations can make comparison of the SCAPS TDS samples (in-situ or ex-situ PV) with the verification samples problematic, and may explain the generally low correlations determined for TCE, DCE and other target analytes at some sites. Results of the in-situ or ex-situ PV samples would not be expected to correlate well with results of the duplicate verification samples where small-scale spatial variation in contaminant concentrations exists. Because of the heterogeneous nature of soil contamination, a considerable amount of uncertainty always exists in comparing results of co-located soil samples, even when obtained from subsamples from a single soil core, and particularly when obtained from a horizontally offset push hole.

#### *4.8.1 The Probabilities of False Positive (FP) and False Negative (FN) Results*

Accuracy of field screening methods is typically measured in terms of the percentage of false positives and false negatives. With ITMS, a false positive result may occur when there are VOCs present in the sample which yield molecular ions or ion fragments with the same  $m/z$  values as the characteristic ions of the target VOCs.

In general, false negatives and positives are determined with respect to a regulatory threshold or standard, above which there is a concern to public health. In the case of VOC-contaminated soils, there are no generally applicable regulatory standards that can be applied. In California, as in other states, cleanup levels are determined on a site-specific basis, considering the specific potential routes of exposure and risks posed by the contamination at that site. For this certification evaluation, since there was no generally applicable standard, false positives and negatives were determined with respect to the detection limits for the TDS analyses and the corresponding verification samples. Rules for determining confirmed positives and negatives, and false positives and negatives were as follows:

*Confirmed Positive (+/+).* In confirmed positives, the TDS and verification sample analyses detected an analyte above their respective detection limits. Unconfirmed positive TDS results occurred where the analyte was detected with the TDS method, but below the detection limit of the verification sample analysis. Unconfirmed positive results were excluded from evaluation of the technology's performance.

*Confirmed Negative (-/-).* In confirmed negatives, either the analyte was not detected with the TDS method nor the verification sample analysis, or was detected with the verification analysis below the detection limit of the TDS method.

*False Positive (+/-).* In false positives the analyte was detected with the TDS method but was not found to be present in the verification sample above the detection limit of the TDS method.

*False Negative (-/+).* In false negatives, the analyte was not detected with the TDS method but was found to be present in the verification sample above the detection limit of TDS method.

Section 7 summarizes the percentage false negative and false positive results achieved for the field studies reviewed for this certification evaluation. Based on the results and reported detection levels for the TDS ex-situ PV samples and EPA Method 8260A, the SCAPS TDS method correctly

reported the presence or absence of TCE, DCE, vinyl chloride, and toluene better than 95% of the time with less than 5% false positives and less than 5% false negative results.

## 5. PREVIOUS FIELD STUDIES

In developing the SCAPS TDS technology a number of field studies had been conducted prior to the evaluation to demonstrate as well as to improve the technology. During this development stage, the TDS samples collected and concentrated on the sorbent traps were analyzed using an on-board GC/PID. The on-board GC/PID was later replaced by an ITMS when this analytical instrument became available for field application through development efforts of Oak Ridge National Laboratory personnel (ORNL). ORNL modified commercially available Finnigan MAT or Teledyne ITMS's for field use with the SCAPS technologies, and designed several direct sampling modules for use with these instruments. Initially the desorption module and sorbent traps used for sample collection were prepared by ORNL but because of desorption and sensitivity issues were replaced with the commercially available OI Analytical purge-and-trap sample concentrator and OI No. 9 traps.

During the period from May 1995 through June 1996 field investigations were conducted at several sites, including Dover AFB, Aberdeen Proving Grounds and Elgin AFB. These field studies involved more than 85 TDS discrete soil sample analyses taken from 32 CP push holes. A summary of these previous investigations is presented below <sup>(5,6,7,8,9)</sup>.

**Table 1. SCAPS TDS - Previous Field Studies Conducted**

Site/Location	TDS Sample Analysis	Dates	No. TDS Pushes	No. Confirmation Samples	Reported Analytes	EPA Method 8260 Confirmation Sample max. conc.(ug/g)	max depth/ lithology
1. Dover AFB	GC/PID & GC/MS	May-95	3	8	cis 12DCE 111TCA TCE PCE ethylbenzene, total xylenes toluene unresolved HCs	2.6 140 150  8 12 2.5 1975525	11 ft. sandy soils
2. Aberdeen Proving Ground	GC/PID; GC/MS for 111TCA	Aug-95	4	9	11DCE 111TCA TCE Toluene	<.014 0.02 0.54 0.006	19 ft. muds, cobbles
3. Elgin AFB	ITMS	May-96	7	4	toluene chlorobenzene T-xylene/ethyl benzene	2.1 <5 50.4	38 ft. sandy soil limestone?
4. Bush River Area, Aberdeen Proving Gound <sup>(1)</sup>	ITMS	Jun-96	18	64	MeCl 1122TCA 112TCA chlorobenze CCL4 TCE	all ND or "J" values, except 1 confirmation sample w/ 0.8 ug/kg 1122TCA	34 ft clay, silt, sands mixtures

(1) Summary report states 64 discrete TDS sample analyses, but is unclear if the same or fewer number of confirmation samples were analyzed

The analytical data from these sites were not directly used in the technology evaluation for several reasons, primarily because of the development stage of the technology when these studies were conducted. During these studies procedures were being modified and for several of the studies the ITMS was not used for the analyses. Additionally, the range of contaminant concentrations found at some sites was quite low.

## 6. FIELD STUDIES CONDUCTED FOR CERTIFICATION EVALUATION

The primary objectives of the field studies were to evaluate: (1) the performance of the SCAPS TDS compared to conventional sampling and analytical methods; (2) the data quality; and (3) the range of technology applications. The secondary objectives were to evaluate the reliability and the potential matrix effects that may affect the performance of the technology. Criteria for selecting the sites for the field studies included: (1) site topography and subsurface conditions which would allow the use of a cone penetrometer; and (2) data from previous investigations which indicate subsurface contamination levels of target VOCs ranging from low ng/g ( $\mu\text{g/kg}$ ) to low  $\mu\text{g/g}$  (mg/kg).

For the certification evaluation U.S. Army Engineer Research and Development Center, Waterways Experiment Station (ERDC-WES) conducted field studies at four different sites with different site conditions and contaminant concentrations. Detailed QA/QC data packages were submitted for field studies completed at three of the sites: Hanover, Lake City and Longhorn. The DTSC Office of Pollution Prevention and Technology Development (OPPTD) was involved up-front in the review of field demonstration plans prior to conducting the field work for the Davis, Lake City and Longhorn sites. Only summary data was submitted for the field study completed at the Davis Transmitter site because of data quality concerns with the non-commercial sorbent traps used in this study. OPPTD observed operations and technology validation procedures in the field during the demonstrations conducted at the Davis and Longhorn sites. For purposes of quality control OPPTD staff obtained duplicate confirmation samples from the Davis and Longhorn sites for analysis by the DTSC Hazardous Materials Laboratory. A summary of the scope of these field investigations is given in table below.

**Table 2.** SCAPS TDS - Field Studies Conducted for Certification Evaluation

Site/Location	TDS Sample Analysis	Dates	No. TDS Pushes	No. Confirmation Samples	Reported Analytes	EPA Method 8260 Confirmation Sample max. conc.(ug/g)	max depth/ lithology
1. Hanover	ITMS	Jun-97	8	33	DCE TCE	4.6 11	60 ft. silt/sandy silts
2. Davis Global Communications Site	ITMS	Dec-97	8	21	PCE	0.09	36 ft. silts/clays/sands
3. Lake City	ITMS	Jul-98	5	16	DCE VC PCE Toluene Ethylbenze	4.7 2.7 0.3 0.3 0.02	13 ft. alluvial deposits
4. Longhorn	ITMS	Aug-98	8	27	TCE DCE	80 2.2	18 ft. sandy clays
<b>TOTAL</b>			<b>29</b>	<b>97</b>			

The four studies involved a total of 29 cone penetrometer push holes which included 97 TDS in-situ discrete soil sample analyses and corresponding number of co-located confirmation samples collected and analyzed by the reference method. Target analytes detected in significant concentrations during the four field studies were tetrachloroethene, trichloroethene, total dichloroethene, vinyl chloride, toluene and ethylbenzene. Detailed results of the SCAPS TDS and verification sample analyses are presented in Appendix I.



## 6.1. Evaluation Procedures

ERDC-WES conducted the field validation studies at the above four sites with assistance from ORNL personnel in the operation of the ITMS and related analytical instrumentation.

### 6.1.1 Verification Samples

The general method for evaluating TDS performance was to compare results of in-situ TDS discrete sample analyses with results of co-located soil core samples analyzed by EPA Method 8260A or other reference method. First, a vertical sequence of discrete in-situ sample analyses with the SCAPS TDS was completed with a cone penetrometer push hole in a zone of known contamination. Verification samples were then obtained from an adjacent, horizontally off-set push hole using the cone penetrometer equipped with a Mostap or ARA core sampler. Each 18-inch long core sample was taken such that the center of the core sample would be located at the same depth below ground surface as the corresponding SCAPS TDS sample. Subsamples, weighing approximately 5 grams, were obtained from the core sample and immediately preserved in methanol using the method by Hewitt<sup>(27)</sup>. Two of the subsamples served as duplicate verification samples and were shipped for off-site laboratory analysis by EPA Method 8260A. These subsamples were taken approximately 8 and 10 inches below the top of the 18-inch core sample. Comparison of the results of the TDS analyses with the average of the results of duplicate verification sample analyses served as the primary basis for evaluating the performance of the SCAPS TDS technology.

### 6.1.2 Additional Soil Sample Analyses by Reference Methods

In addition to the duplicate verification samples, additional 5-gram subsamples were taken along the length of the core and immediately preserved in methanol using the method by Hewitt<sup>(27)</sup>. These samples were shipped off-site for analysis using EPA Method 8260A or other reference method. These samples were to provide an indication of vertical variation or heterogeneities in contaminant concentration which could potentially explain differences between results of the SCAPS TDS analyses and the verification samples. In addition, two samples located above and below the duplicate verification samples were analyzed for moisture content and density. A review of results related to these physical soil properties was not part of the evaluation.

### 6.1.3 Ex-situ TDS Probe Verification (PV) Samples

A subsample of approximately 5 grams was obtained from the center of each core sample (9 inches from the top of the core) and sized such that it could be injected directly into TDS Probe sample chamber. The depth of the PV sample was to correspond exactly to the depth of the corresponding in-situ TDS discrete sample. With the TDS probe out of the ground or “ex-situ”, the PV sample “plugs” were injected into the TDS Probe sample chamber for thermal desorption and analysis by ITMS.

Results of the PV sample analyses were used for several purposes. First, the PV sample result was used to establish that the target contaminant concentration encountered in the TDS push hole at that sampling depth was the same or similar to that encountered in the horizontally off-set verification sample push hole. If concentration levels were very different, then comparison of the TDS result with the verification sample result at that depth would not be appropriate. Such a result

would be an indicator of high spatial variation or heterogeneity in contaminant concentrations at that sampling depth.

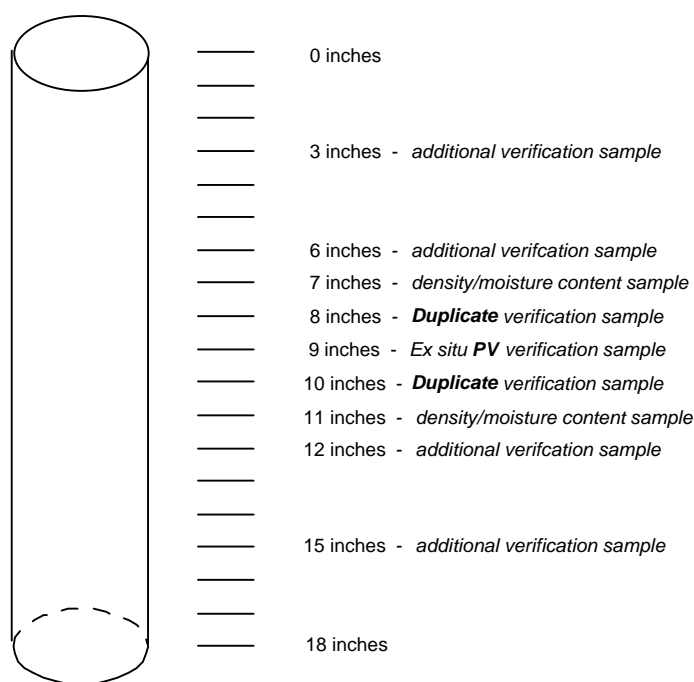
Second, a direct comparison of the results of ex-situ PV sample with the duplicate verification samples would provide another means, or at least a check, to assess performance of the technology. The PV sample was taken from the verification core sample as a subsample located adjacent and in between the two subsamples taken for the duplicate verification samples. Because these subsamples were taken from the same core sample from a single push hole, it represents the best case for comparing TDS results with the reference method. However, because the TDS analysis for the PV sample was ex-situ, and not in-situ as would be the case in a field application of the technology, this analysis by itself was not considered sufficient to validate the technology.

Lastly, the PV samples were used to evaluate whether the TDS probe effectively desorbed VOCs from the soil samples. This was accomplished by analysis of the PV sample for any VOC after sampling was completed. After each PV sample was expelled from the sample chamber it was preserved in methanol and analyzed by EPA Method 8260A along with the verification samples.

#### 6.1.4 Core Subsampling Scheme Layout

Figure 2, below shows approximate locations and types of subsamples which were obtained from each collected verification core sample.

**Figure 2**  
Verification Subsampling Scheme Along  
Typical 18 - Inch Core Sample





### 6.1.5 Laboratory Analysis

Verification samples were preserved with methanol in the field by the method of Hewitt<sup>(27)</sup> and EPA method 5035<sup>(18)</sup> and, except for the Hanover site field study, all samples were analyzed by GCMS using EPA Method 8260A.

The U.S. Army Engineer Research and Development Center, Environmental Chemistry Branch (ERDC-ECB), Vicksburg, Mississippi analyzed the methanol-preserved verification samples using EPA Method 8260A. A small percentage of splits of verification samples were analyzed by a second laboratory, PDP Analytical Services, certified by the US Army Corps of Engineers Hazardous, Toxic and Radioactive Waste Program. These provided an external QC check on the verification data produced by the ERDC-ECB laboratory. Additionally, the DTSC Hazardous Materials Laboratory analyzed selected verification samples for QA/QC purposes.

For the Hanover Site Field Study, the U.S. Army Corp of Engineers Cold Regions Research Laboratory analyzed additional methanol preserved sub-samples taken along the length of the verification core samples using EPA Method 5021<sup>(25)</sup>, an equilibrium headspace analysis of VOCs in soils using gas chromatography. These additional samples were analyzed to evaluate the vertical variation in contaminant concentration along the length of the core sample.

## 6.2 Hanover

The SCAPS TDS field demonstration at the Cold Regions Research and Engineering Laboratory (CRREL) in Hanover, New Hampshire was conducted between June 2 and June 14, 1997. The SCAP TDS analyses were performed using a Finnigan ITMS. The area was the site of a former TCE spill and the primary contaminants and target analytes were TCE and DCE. Soil types encountered at the site consisted of silts embedded with layers of fine sandy silt. The sandy silt layers ranged from less than 1 inch to several feet.

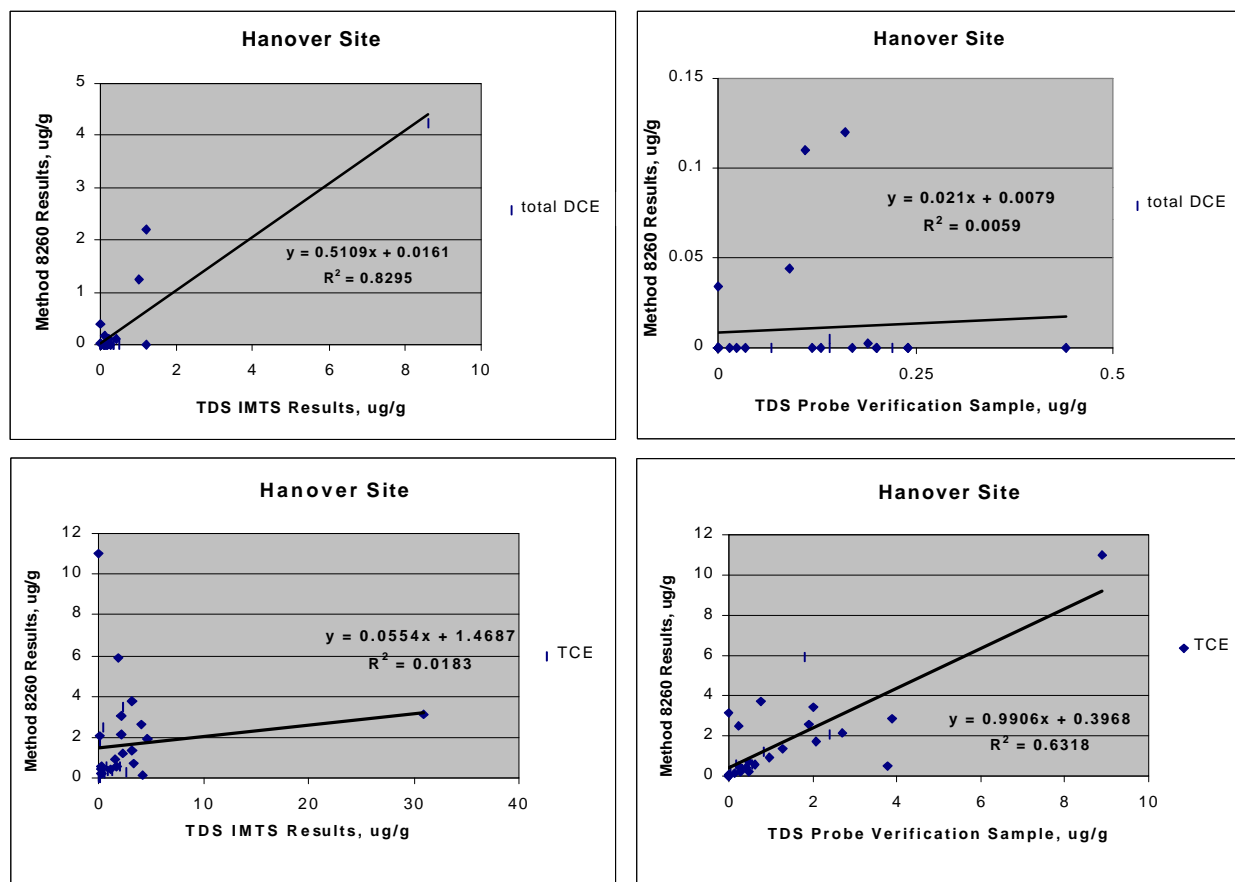
For this demonstration, cone penetrometer push holes were completed at 5 different locations to depths of up to 60 feet to obtain in-situ discrete soil analyses using the SCAPS TDS technology. Verification samples were obtained using a Mostap core sampler driven with the cone penetrometer at a co-located push-hole which was horizontally off-set from the SCAPS TDS push hole by 8 to 12 inches. Verification samples and ex-situ PV samples were obtained by subsampling along the Mostap core as discussed in Section 6.1. Additional subsamples were taken along the Mostap core samples for analysis by EPA Method 5021, a headspace GC method to provide data on the vertical distribution of contaminants.

At 4 of the 5 locations a number of the SCAPS TDS analyses and verification samples (including push holes) were repeated because of poor comparisons with the verification samples or losses of the bottom portion of the Mostap core sample (need to review this issue further). A total of 35 TDS in-situ discrete soil sample analyses for TCE and DCE were completed for which 33 corresponding verification sample results by EPA Method 8260A were obtained. A push hole at a sixth location was completed to assess the application of SCAPS TDS in relatively high contaminant concentrations. In this case only an ex-situ PV sample and corresponding verification sample were obtained. A number of PV sample results from the fifth push hole were excluded from the analysis due to high PV sample residue results, suggesting that all of the contaminants were not thermally desorbed from the sample.

Plots of the SCAPS TDS results for TCE and DCE versus the average of the corresponding duplicate verification samples are presented below. Also plotted below are the probe verification

sample results versus the verification sample results. The regression lines and correlation coefficients ( $R^2$ ) are presented only for informational purposes. Such an analysis may not be that meaningful because of the small-scale spatial variation and the relatively low and narrow range of contaminant concentrations encountered at the Hanover site.

**Figure 3**  
Hanover Site - Results for Total DCE and TCE  
SCAPS TDS vs. EPA Method 8260A



The performance of the SCAPS TDS technology at the Hanover site was evaluated only in terms of the numbers of false positive and false negatives with respect to the results of EPA Method 8260A validation samples. For the PV sample results, 5% or less of the total number of TCE or DCE analyses were found to be either false positives or false negatives. In comparing the SCAPS TDS in-situ TCE results with the validation samples results there were 94% true positives and negatives versus 6% false negatives. The 6% represented two false negative results, one of which appears to be due to soil heterogeneity encountered at the sampling location. These results are presented in the table below.

**Table 3**  
Hanover Site - False Positive / False Negative Results  
SCAPS TDS vs. EPA Method 8260A Verification Sample Results

Number of Samples/ Percent of Total Samples		TRUE		FALSE		Total
		POS (+/+)	NEG -/-	POS +/-	NEG -/+	
TCE	in-situ TDS	28 88%	2 6%	0 0%	2 6%	32 100%
	ex-situ PV	27 84%	4 13%	0 0%	1 3%	32 100%
DCE	in-situ TDS	8 38%	9 43%	3 14%	1 5%	21 100%
	TDS ex-situ PV	5 26%	13 68%	1 5%	0 0%	19 100%

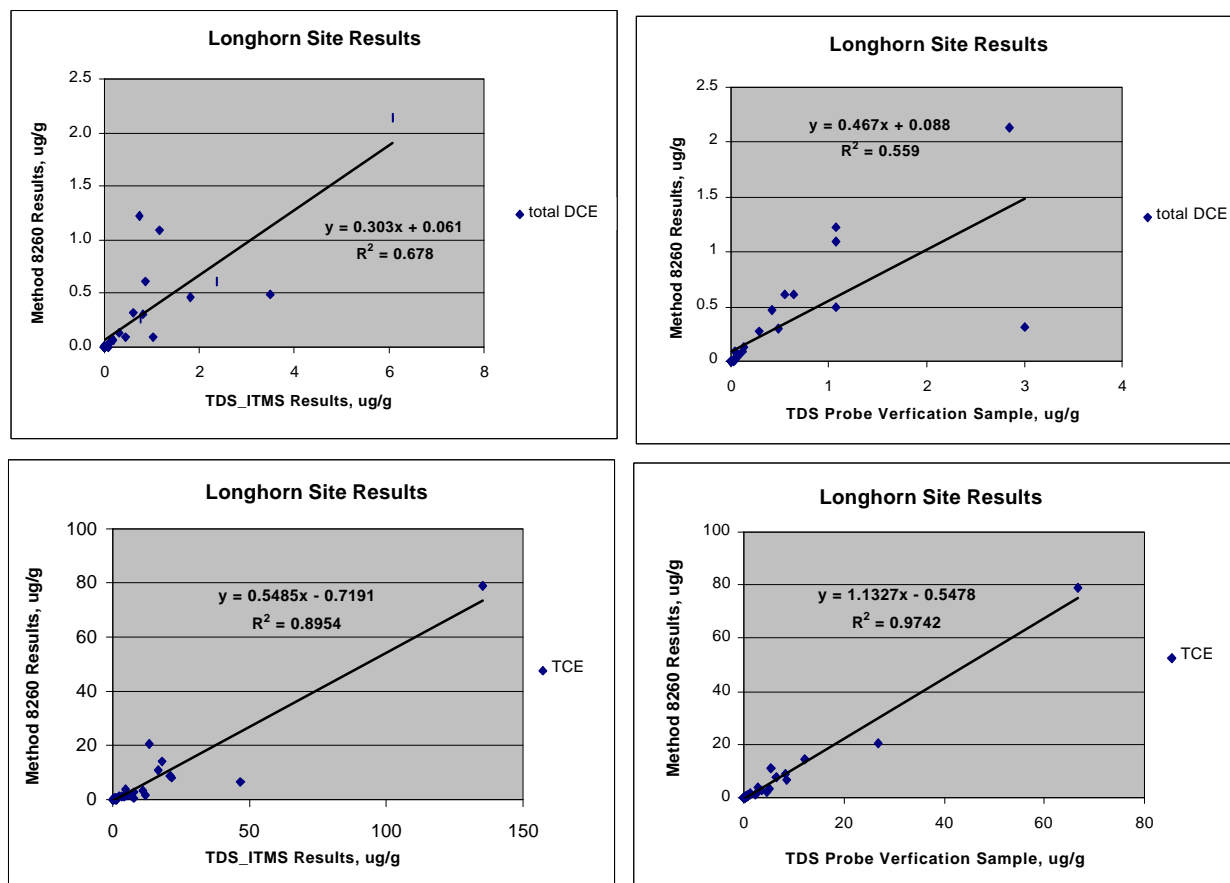
### 6.3 Longhorn

The SCAPS TDS field demonstration at the Longhorn Army Ammunition Plant in Karnack, Texas was conducted between August 10 and August 21, 1998. Two areas were investigated. The first was an area along a street across from an old jet engine testing facility which was known to have TCE groundwater contamination. The second was an area in front of an equipment facility which had used solvents. Depth to groundwater at the Longhorn site varies from 5 to 20 feet. The underlying sediments encountered during the investigation consisted of medium plasticity sandy clays with some zones of higher plasticity to a depth of 4 to 10 feet. For the field demonstration 8 cone penetrometer push holes were completed to depths of up to 18 feet to obtain 27 in-situ discrete soil sample analyses with the SCAPS TDS technology. Co-located verification samples were obtained from adjacent push holes (horizontally off-set by 8 to 12 inches) using an ARA core sampler driven with the cone penetrometer. Verification samples for analysis by EPA Method 8260A and PV samples for ex-situ SCAPS TDS analyses were obtained by subsampling along the ARA core sample as discussed in Section 6.1. The target analytes and primary contaminants detected at the site were TCE and total DCE.

Plots of the SCAPS TDS results for TCE and DCE versus the average of the corresponding duplicate verification samples are presented below. Also plotted below are the PV sample results versus the verification sample results. Again, the regression lines and correlation coefficients ( $R^2$ ) are presented for informational purposes only.

Performance of the SCAPS TDS was evaluated in terms of the numbers of false positive and false negatives with respect to the results of EPA Method 8260A verification samples. These results, presented in the table below, indicate that the SCAPS TDS technology performed well. Both the SCAPS TDS and PV sample results had less than 5% false positives or negatives for TCE and DCE when compared to the EPA Method 8260A validation sample results.

**Figure 4**  
Longhorn Site Results for Total DCE and TCE  
SCAPS TDS vs. EPA Method 8260A



**Table 4**  
Longhorn Site - False Positive / False Negative Results  
SCAPS TDS vs. EPA Method 8260A Verification Sample Results

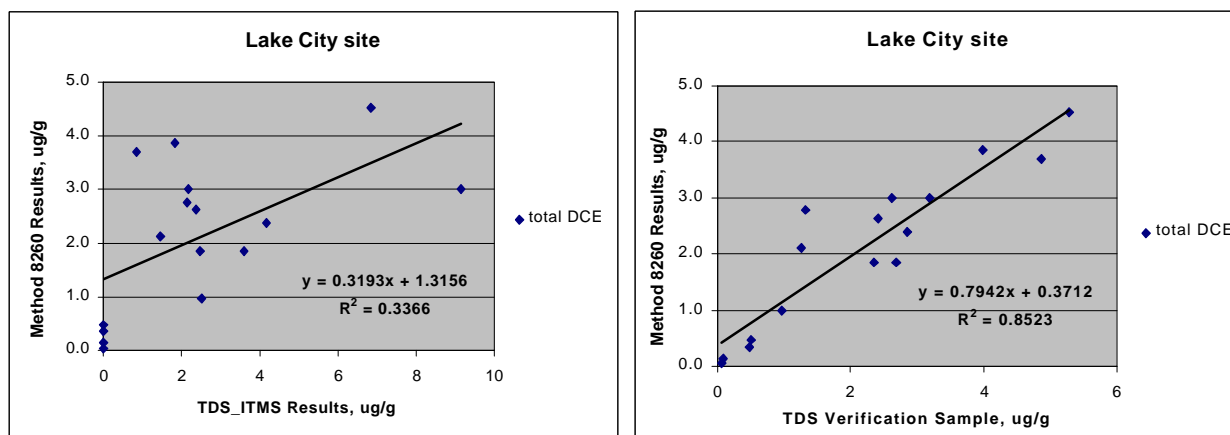
Number of Samples/ Percent of Total Samples		TRUE		FALSE		Total
		POS (+/+)	NEG -/-	POS +/-	NEG -/+	
TCE	in-situ TDS	27	0	0	0	27
		100%	0%	0%	0%	100%
	ex-situ PV	28	0	0	0	28
		100%	0%	0%	0%	100%
DCE	in-situ TDS	16	10	1	0	27
		59%	37%	4%	0%	100%
	TDS ex-situ PV	18	9	0	0	27
		67%	33%	0%	0%	100%

## 6.4 Lake City

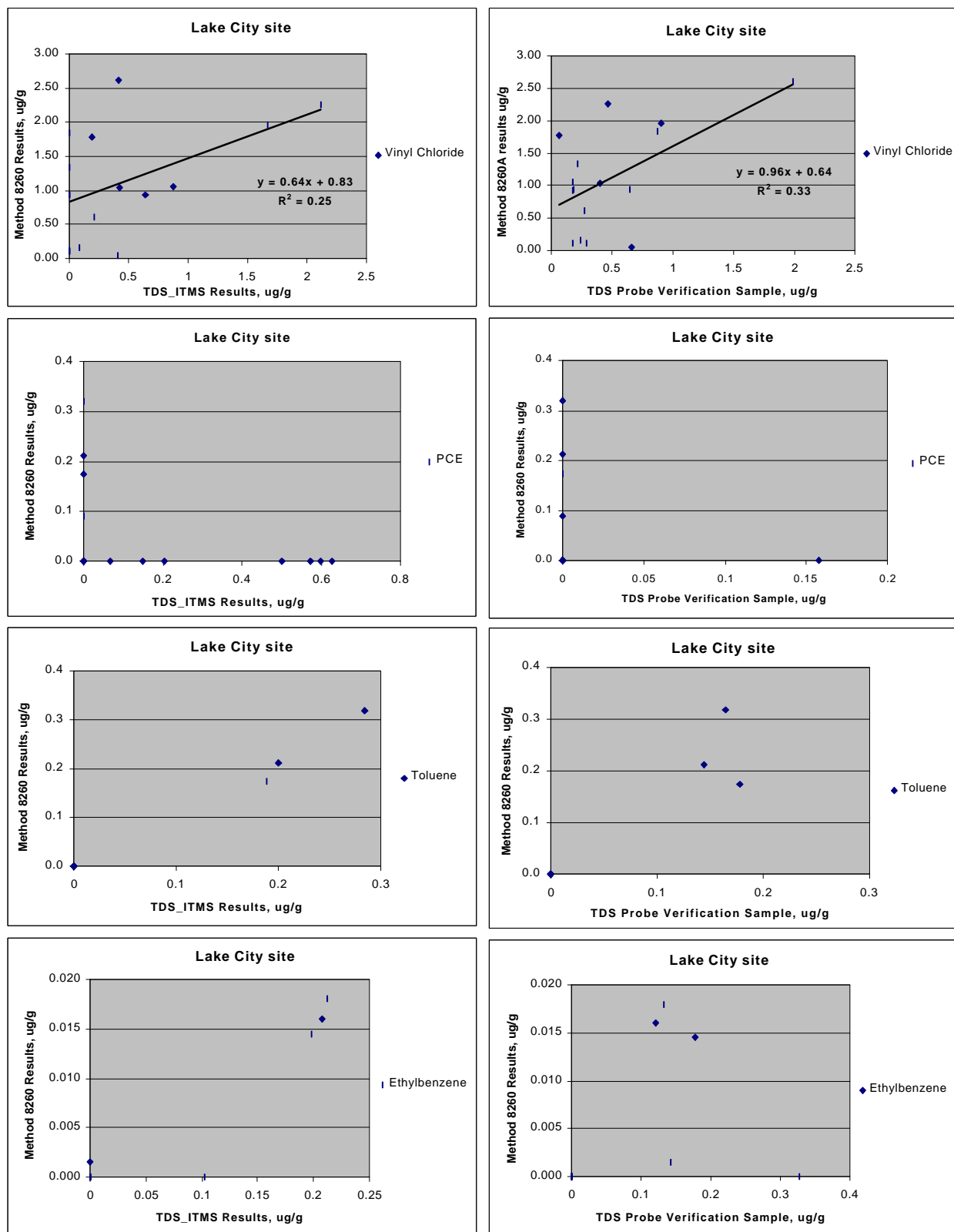
The SCAPS TDS field demonstration was conducted between July 6 and July 17, 1998 at the old oil and solvent pits site located in Area 17 of the Lake City Army Ammunition Plant in Lake City, Missouri. SCAPS TDS pushes were completed along a dirt road adjacent to the central pit. The unlined pit was used since the 1960's for the disposal of waste grease and oils, and solvents. Contaminants previously identified at this site included, dichloroethane, dichloroethene, trichloroethane, trichloroethene, tetrachlorethane, ethylbenzene, and toluene. The site geology consisted of alluvial deposits with depth to groundwater ranging from 5 to 20 feet. The field demonstration consisted of completing 5 cone penetrometer push holes to depths of up to 13 feet to obtain 16 in-situ discrete soil sample analyses with the SCAPS TDS technology. Co-located verification samples were obtained from adjacent push holes (8 to 12 inches horizontally off-set) using an ARA core sampler driven with the cone penetrometer. Verification samples for analysis by EPA Method 8260A and PV samples for ex-situ SCAPS TDS analyses were obtained by subsampling along the ARA core sample as discussed in Section 6.1. For the Lake City demonstration the ITMS was set up to analyze for seven target compounds: vinyl chloride, total DCE, TCE, PCE (tetrachloroethene), benzene, toluene and ethylbenzene. The ITMS was calibrated for all of these compounds except benzene.

Plots of the SCAPS TDS results for the target analytes versus the average of the corresponding duplicate verification samples are presented below. Also plotted below are the probe verification sample results versus the verification sample results. The regression lines and correlation coefficients ( $R^2$ ) are presented for informational purposes, and only for vinyl chloride and total DCE results. For PCE, toluene and ethylbenzene the concentrations encountered were around the limits of detection and there were few confirmed positives.

**Figure 5**  
Lake City Site Results for Total DCE  
SCAPS TDS vs. EPA Method 8260A



**Figure 6**  
Lake City Site Results for Vinyl Chloride, PCE, Toluene and Ethyl Benzene  
SCAPS TDS vs. EPA Method 8260



To evaluate technology performance, analyte concentrations over at least two orders of magnitude is desirable. For several of the target analytes concentrations encountered were around the limit of detection and there were few confirmed positive results. For these analytes, PCE, toluene and ethyl benzene, the data generated at this site were insufficient to adequately evaluate technology performance. For vinyl chloride, there were a number of confirmed positives, but concentration levels were low, all results less than 2 µg/g for the verification samples. For total DCE, verification sample concentrations were higher and ranged up to 5 µg/g. For DCE there was relatively good correlation between the PV sample and verification sample results ( $R^2=0.85$ ).

The number and percent false positive and false negative results for the in-situ TDS and ex-situ PV sample analyses are presented in the table below.

**Table 5**  
Lake City Site - False Positive / False Negative Results  
SCAPS TDS vs. EPA Method 8260A Verification Sample Results

ANALYTE	TRUE		FALSE		Total
	POS (+/+)	NEG -/-	POS +/-	NEG -/+	
In-Situ TDS Sample Results					
DCE	12	1	0	3	16
	75%	6%	0%	19%	100%
Vinyl Chloride	10	2	0	4	16
	63%	13%	0%	25%	100%
PCE	0	6	5	3	14
	0%	43%	36%	21%	100%
Toluene	3	13	0	0	16
	19%	81%	0%	0%	100%
Ethyl Benzene	3	12	0	0	15
	20%	80%	0%	0%	100%
Ex-Situ PV Sample Results					
DCE	16	0	0	0	16
	100%	0%	0%	0%	100%
Vinyl Chloride	16	0	0	0	16
	100%	0%	0%	0%	100%
PCE	0	12	1	3	16
	0%	75%	6%	19%	100%
Toluene	3	13	0	0	16
	19%	81%	0%	0%	100%
Ethyl Benzene	4	11	1	0	16
	25%	69%	6%	0%	100%

A major concern with analytical field screening technologies is the potential for false negatives, not detecting a contaminant when it is actually present above the method detection limit. Based on the results of ex-situ PV sample analyses there were no false negatives except for PCE. For the in-situ TDS analyses there were relatively large percentages of false negatives for vinyl chloride, DCE and PCE, and a high percentage of false positives for PCE only. The in-situ TDS false negatives for DCE may be explained by the differences in the PV sample result from the

corresponding in-situ TDS result, indicating a concern with soil heterogeneity at that sampling location. For the 4 in-situ TDS false negatives encountered for vinyl chloride in a single TDS push hole, the analyst's log noted that the quantification ion for vinyl chloride was present, but below the reportable detection limit. For the low concentrations of vinyl chloride encountered, the verification sample results generally appear higher than either the in-situ TDS or ex-situ PV sample results.

The high numbers of false negatives and positives for PCE could not be explained. There were no confirmed positive results for either the in-situ TDS or PV sample results. At some sampling locations PCE was detected only in the in-situ TDS samples. Of concern are the results where the verification samples detected PCE while the associated PV sample did not. Because of the low PCE contamination levels encountered, generally less than 0.4 µg/g, it was not possible to adequately evaluate performance for this analyte.

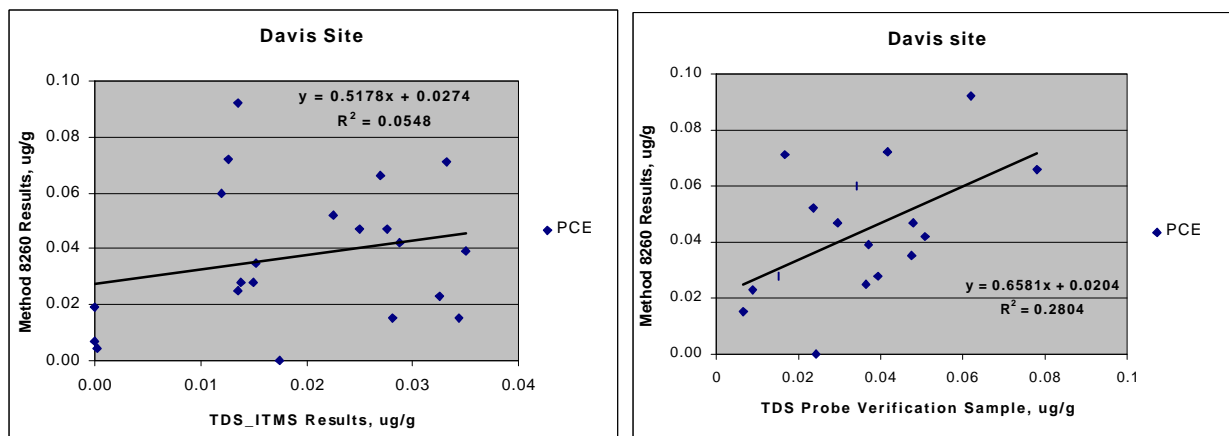
## 6.5 Davis Global Communication Site

Initially the SCAPS TDS field demonstration was planned to be conducted at the Investigation Cluster 33 site located at McClellan AFB, where previous investigations had identified significant levels of chlorinated solvent contamination in a relatively deep vadose zone. Groundwater at this location occurs at depths greater than 80 feet below ground surface. After several cone penetrometer attempts were unsuccessful in penetrating below hardpan at 5 to 13 feet below ground surface, the field work was moved to the Global Communications Site. This problem illustrates the limitation in the use of CP technology at some sites.

The Davis Global Communication Site is an annex of McClellan Air Force Base located 4 miles south of Davis, California. In 1985, three underground storage tanks containing diesel fuel were found to be leaking. During the field investigation for hydrocarbon contamination, chlorinated VOC contamination from an unknown source was discovered in the vadose zone and groundwater. Previous investigations detected VOC concentrations ranging up to 1.38 mg/l in groundwater monitoring wells.

The field demonstration consisted of completing 8 cone penetrometer push holes to depths of up 36 feet to obtain 21 in-situ discrete soil sample analyses with the SCAPS TDS technology. Plots of the SCAPS TDS and PV sample results for PCE versus the average of the corresponding duplicate verification samples are presented below. As before, the regression lines and correlation coefficients ( $R^2$ ) are presented for informational only.

**Figure 7.** Davis Site Results for Tetrachloroethene - SCAPS TDS vs. EPA Method 8260A





The primary contaminant detected, PCE, was found at very low concentrations, less than 0.01 µg/g. These reported quantification levels are generally lower than the detection levels achieved at the Hanover, Longhorn or Lake City sites. Generally for these sites most detection limits reported were above 0.1 µg/g, although 0.05 µg/g detection limits were reported for a few analyses. Since only summary results were reported, the quantitation levels reported for Davis this site could not be confirmed.

Based on the summary results reported there were no false negatives for the PV sample results. For the in-situ TDS results, 2 of 21 results or less than 5 percent were false negatives. For these two samples results, PCE was detected in the corresponding PV samples, indicating that soil heterogeneity at this sampling location might explain the false negative results.

A Finnigan ITMS was used for the Davis demonstration in conjunction with a thermal desorption unit and non-standard traps provided by ORNL. These traps were found to be non-reproducible, biasing the data. After discovering this problem, the system was changed back to a more proven sample introductory system for the ITMS and industry standard traps (the OI analytical purge-and-trap unit and standard Tenax and OI Analytical No. 9 traps). Because of this problem, the Davis site data are not considered representative of the TDS's capabilities

## **6.6 Quality Assurance / Quality Control Review**

The TDS system was purged and a blank sample was introduced to evaluate the cleanness of the system prior to the daily operation. TDS performance was evaluated daily by injecting a 250 ng gas mixture containing DCE, TCE, and ethylbenzene (PCE and xylene were included in the Hanover study). The percent recoveries of each analyte were recorded. The recoveries of DCE were generally low for studies conducted at the Lake City and Longhorn sites but appear to be acceptable for the Hanover site. The lower recoveries for DCE were the result of higher ambient temperatures encountered at the Lake City and Longhorn sites. Morning temperatures in New Hampshire were usually in the 60 °F range or lower, thus the gaseous DCE stayed liquid and could be pulled up into the syringe for dispensing. In Missouri and Texas in July and August, the morning temperatures were much higher and proved a hindrance to dispensing an accurate volume of neat DCE into the Tedlar bag. ITMS performance was evaluated by daily calibration using a pure standard mixture with concentrations ranging from 25 to 750 ng incorporated in the analytical series to monitor the precision and accuracy of the analytical system over the course of the day.

For QA/QC purposes, splits of selected verification samples were analyzed by an independent laboratory. DTSC Hazardous Materials Laboratory staff reviewed the data packages for the results of these quality control samples<sup>(29)</sup> which were analyzed by PDP Analytical Services for the Hanover site, and Argus Analytical for the Lake City and Longhorn sites. Not all the control limits were met on one of the Hanover site QC samples (ID#72996) and holding times on the QC samples for the Longhorn site were not met. Results for QC samples for the Lake City site were found to be acceptable.

## 7. SUMMARY OF RESULTS

Field verification data was collected on six analytes at hazardous waste sites in four different geographic areas. A total of 29 SCAPS TDS pushes were completed to obtain 97 separate TDS in-situ discrete soil sample analyses using the on-board ITMS along with a corresponding number of co-located confirmation samples which were collected and analyzed by the reference method. For two of the target analytes investigated, TCE and total DCE, approximately 60 TDS analyses spanning a range of concentrations were obtained and verified. There were 16 TDS analyses completed at the Lake City site which provided limited data on other analytes including PCE, vinyl chloride, toluene and ethyl benzene.

The table below summarizes the maximum concentrations of analytes detected during each of the field studies and their associated detection limits. Included are results for the in-situ TDS analyses and ex-situ PV sample analyses, as well as the EPA Method 8260A analyses. The performance of an analytical method is typically evaluated over a range of concentrations of two or more orders of magnitude. The maximum concentration encountered for any of the analytes was 131  $\mu\text{g/g}$  for TCE at the Longhorn site. Except for TCE, the concentrations encountered during the field studies were relatively low. For vinyl chloride and DCE, concentrations ranged up to 2  $\mu\text{g/g}$  and 9  $\mu\text{g/g}$  respectively. For the other analytes, only low concentrations in the region of the detection limits were encountered. The detection limits achieved with the SCAPS TDS method were comparable to those of EPA Method 8260A, the reference method. Reported detection limits for all of the analytes were similar for both TDS and reference method analyses, with median values ranging from about 0.1 to 0.3  $\mu\text{g/kg}$ .

**Table 6**

Maximum Concentrations and Median Detection Limits for Analytes Investigated

Analyte	Site	Maximum Concentration ( $\mu\text{g/g}$ )			Reported (Median) Detection Limit ( $\mu\text{g/g}$ )		
		TDS	TDS-PV	8260	TDS	TDS-PV	8260
DCE	Hanover	8.6	1.2	3.9	0.156	0.103	0.364
	Longhorn	6.07	3	2.54	0.200	0.067	0.120
	Lake City	9.15	5.278	5.16	0.125	0.140	0.103
TCE	Hanover	31	8.9	11	0.156	0.109	0.367
	Longhorn	135	66.7	80.1	0.330	0.089	0.120
PCE	Lake City	0.628	0.158	0.35	0.103	0.285	0.126
	Davis	0.035	0.078	0.092	NR	NR	NR
VC	Lake City	2.12	1.99	3.25	0.125	0.140	0.103
Toluene	Lake City	0.28	0.18	0.353	0.125	0.184	0.102
Ethylbenzene	Lake City	0.212	0.327	0.03	0.134	0.186	0.102

Since the SCAPS TDS is considered a field screening technology, performance was primarily evaluated in terms of the potential for false positive and false negative results. Occurrences of false positives and negatives, as well as confirmed positives and negatives, were determined based on results for the in-situ TDS analyses and ex-situ TDS PV sample analyses versus the analysis by reference method, EPA Method 8260A. For field screening technologies, performance is generally considered acceptable if there are fewer than 5% false negatives and fewer than 5% false positives. Of particular concern is the occurrence of false negatives, that is the event of determining a sample

is clean or uncontaminated when it is not. These results for the four sites studied are summarized in the two tables below.

**Table 7**  
Summary of False Positive and Negative Results  
In-Situ TDS Results vs. Method 8260A Verification Sample Results

Analyte	Site	# Borings	Number TDS Analyses	TRUE		FALSE	
				POS +/-	NEG -/-	POS +/-	NEG -/-
DCE	Hanover	7	21	8	9	3	1
	Longhorn	8	27	16	10	1	0
	Lake City	5	16	12	1	0	3
			<b>64</b> <i>Percent Total =</i>	36 56%	20 31%	4 6%	4 6%
TCE	Hanover	7	32	28	2	0	2
	Longhorn	8	27	27	0	0	0
			<b>59</b> <i>Percent Total =</i>	55 93%	2 3%	0 0%	2 3%
PCE	Lake City	5	14	0	6	5	3
	Davis	8	21	18	0	1	2
			<b>35</b> <i>Percent Total =</i>	18 51%	6 17%	6 17%	5 14%
VC	Lake City	5	<b>16</b> <i>Percent Total =</i>	10 63%	2 13%	0 0%	4 25%
Toluene	Lake City	5	<b>16</b> <i>Percent Total =</i>	3 19%	13 81%	0 0%	0 0%
Ethylbenzene	Lake City	5	<b>15</b> <i>Percent Total =</i>	3 20%	12 80%	0 0%	0 0%

**Table 8**  
Summary of False Positive and Negative Results  
Ex-Situ PV Sample Results vs. Method 8260A Verification Sample Results

Analyte	Site	# Borings	Number TDS Analyses	TRUE		FALSE	
				POS +/-	NEG -/-	POS +/-	NEG -/-
DCE	Hanover	7	19	5	13	1	0
	Longhorn	8	27	18	9	0	0
	Lake City	5	16	16	0	0	0
			<b>62</b> <i>Percent Total =</i>	39 63%	22 35%	1 2%	0 0%
TCE	Hanover	7	32	27	4	0	1
	Longhorn	8	28	28	0	0	0
			<b>60</b> <i>Percent Total =</i>	55 92%	4 7%	0 0%	1 2%
PCE	Lake City	5	16	0	12	1	3
	Davis	8	17	16	0	1	0
			<b>33</b> <i>Percent Total =</i>	16 48%	12 36%	2 6%	3 9%
VC	Lake City	5	<b>16</b> <i>Percent Total =</i>	16 100%	0 0%	0 0%	0 0%
Toluene	Lake City	5	<b>16</b> <i>Percent Total =</i>	3 19%	13 81%	0 0%	0 0%
Ethylbenzene	Lake City	5	<b>16</b> <i>Percent Total =</i>	4 25%	11 69%	1 6%	0 0%

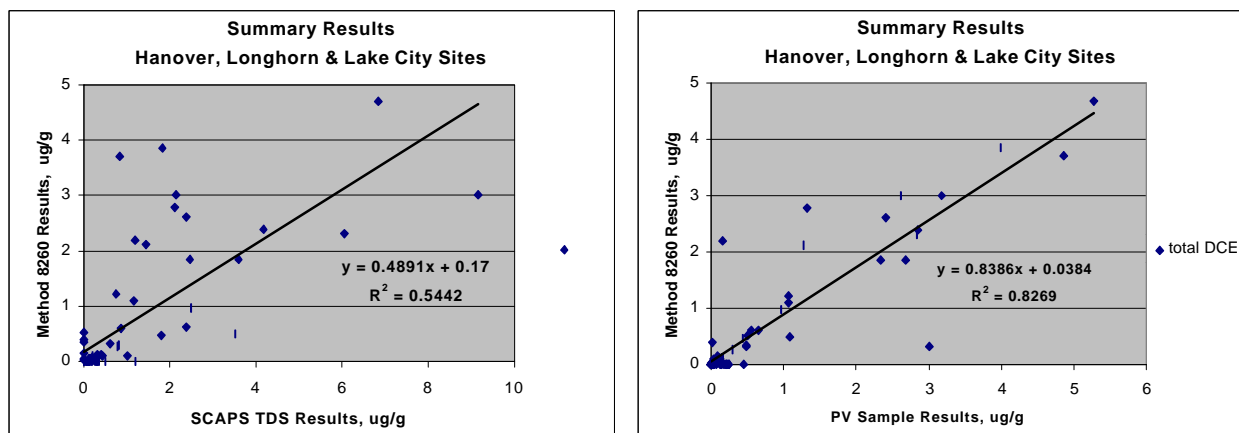
For all of the analytes, there was a greater percentage of false positive and negative results for the in-situ TDS analyses than for the ex-situ PV sample analyses. This was an expected result due to soil contamination variability between the location of the in-situ TDS discrete sample and location of the verification sample obtained from a horizontally off-set push hole. Even given this problem there were only 3% false negatives and no false positives for TCE based on the in-situ TDS results. For DCE there were 6% false negatives and 6% false positives based on the in-situ TDS results which was only 1% higher than the generally accepted performance standard of 5% for field screening technologies. For Both DCE and TCE, there were 2% or fewer false positive or false negative results based on the PV sample results, indicating an acceptable level of performance.

For vinyl chloride, there were 16 confirmed positive results for concentrations of up to 2 µg/g at the Lake City site. There were only several confirmed positive TDS results for low concentrations (< 0.4 mg/kg) of toluene and ethylbenzene at the Lake City site. These few confirmed positive results for toluene and ethylbenzene were not considered sufficient to adequately assess the performance of the technology for these analytes.

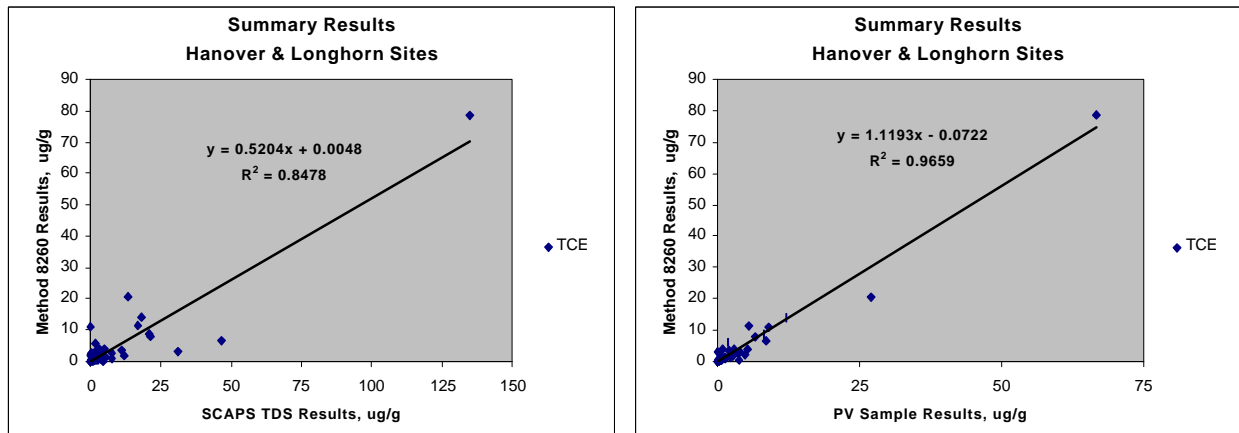
Data on PCE were collected from both the Lake City and Davis sites. However, for the Davis site a supporting data package was not provided, and only low concentrations (0.01 to 0.04 µg/g) of PCE were encountered that were less than the reported detection limits for any of the other sites. Because of this concern and data quality concerns with the use of non-commercial sorbent traps for the Davis site, these results were not considered in the certification evaluation.

Plotted below are the pooled data for DCE from the Hanover, Longhorn and Lake City sites, and the pooled data for TCE from the Hanover and Longhorn sites. For these two analytes having the most performance data there appears to be a relatively good correlation between the TDS PV sample results and verification sample result ( $R^2=0.83$  for DCE;  $R^2=0.97$  for TCE). As expected, the correlations for the in-situ TDS analysis with verification sample results were less due to small-scale spatial variation in contamination levels encountered at the sites ( $R^2=0.54$  for DCE;  $R^2=0.85$  for TCE).

**Figure 8**  
Summary of DCE Results - Hanover, Longhorn and Lake City Sites



**Figure 9**  
Summary of TCE Results for Hanover and Longhorn Sites



## 8. REFERENCES

1. California Hazardous Waste Environmental Technology Certification Program, Eligibility and Acceptance Criteria Guidance and Eligibility Application Form, (Received 9/23/96)
2. Laboratory Evaluation of a Volatile Organic Analysis System for the Characterization and Analysis Penetrometer System, by Karen Myers, James M. Brannon, Richard A. Karn, Cynthia B. Price, Dan Y. Eng, Ann B. Strong, Stafford S. Cooper, US Army Engineer Waterways Experiment Station, Technical Report IRRP-95-3, September 1995.
3. Draft Method 8265, Direct Sampling Ion-Trap Mass Spectrometry for the Measurement of Volatile Organic Compounds In Water, Soil, and Air, Prepared by: Marcus B. Wise, Michael R. Guerin, Roosevelt Merriweather, and Cyril V. Thompson, Oak Ridge National Laboratory, and William M. Davis, U.S. Army Corps of Engineers, Waterways Experiment Station, June 1997.
4. In-Situ Thermal Desorption of VOCs in Vadose Zone Soils, *Field Analytical Chemistry and Technology* 2(3)L163-171, 1998, John Wiley & Sons, Inc. prepared by Karen Myers, Richard A. Karn, Dan Y. Eng, Alan D. Hewitt, Ann B. Strong, and James M. Brannon. M. Davis, John S. Furey, and Beth Porter.
5. Thermal Desorption VOC Sampler Field Trial, Aberdeen Proving Ground; Undated report on second field trial conducted at Aberdeen Proving Ground on August 21-24, 1995.
6. Technology Demonstration Plan, Tri-service Site Characterization and Analysis Penetrometer System (SCAPS) Validation at Bush River Stucy Area, Edgewood Area, Aberdeen Proving Ground, Maryland, May 1996 , Prepared by: US Army Engineer Waterways Experiment Station Vicksburg, MS.
7. Tri-Service Site Characterization and Analysis Penetrometer System Volatile Organic Compound Sensor Validation a Aberdeen Proving Ground Maryland prepared by William Davis, James Brannon, George Robitaille, Jerry Burgess, John Furey, Cyril Thompson, Cynthia Price, Richard Dkarn, Robert Jones, and Karen Myers, undated (describes June 1996 field study).
8. Thermal Desorption Volatile Organic Compound (VOC) Field Sampler Investigation, Elgin AFB, Florida, by Karen Myers, Richard Karn, Cynthia B. Price, Dan Y. Eng, and Ann B. Strong, undated
9. Thermal Desorption VOC Sampler: Improvements and Field Trial Performance (Dover AFB) Draft, undated by Karen F. Myers, James M. Brannon, Richard A. Karn, Cynthia B. Price, Dan Y. Eng, Ann B. Strong, U.S. Army Corps of Engineers, Waterways Experiment Station
10. Final Technology Demonstration Plan, Tri-Service Site Characterization and Analysis Penetrometer System (SCAPS) Validation at McClellan Air Force base, California, Prepared by: US Army Engineer Waterways Experiment Station, Vicksburg, MS, October 1996.
11. Tri-Service Site Characterization and Analysis Penetrometer System Volatile Organic Compound Sensor Validation at McClellan Air Force Base, California by William Davis, James Brannon, Marc

Wise, Karen Myers, George Robitaille, Melissa Ruttel, Tim Chapman, John Furey, Cyril Thompson, Jeff Powell, Dan Eng, Richard Burrows, Karl Konecny, Robert Jones, and Richard Karn; received with February 27, 1997 memorandum from Philip H. Mook, McClellan AFB, transmitting summary of SCAPS technology demonstration conducted at the Davis Transmitter Site in November and December 1996.

12. Technology Demonstration Plan, Tri-Service Site Characterization and Analysis Penetrometer System (SCAPS) Validation at Lake City Army Ammunition Plant, MO, Prepared by: Ms. Karen F. Myers, US Army Engineer Waterways Experiment Station, Vicksburg, MS, July 1998.

13. Final Technology Demonstration Plan, Tri-Service Site Characterization and Analysis Penetrometer System (SCAPS) Validation at Longhorn Army Ammunition Plant, Karnack, TX, Prepared by: Ms. Karen F. Myers, US Army Engineer Waterways Experiment Station, Vicksburg, MS, August 10-21, 1998; Appendices included:

14. Thermal Desorption VOC Sampler Data Package for US Army Corps of Engineers, Cold Regions Research and Engineering Laboratory (CRREL), Hanover, NH, June 2, 1997 - June 14, 1997, submitted by Karen Myers, US Army Engineer Waterways Experiment Station, Vicksburg, MS.

15. Thermal Desorption VOC Sampler Data Package for Lake City Army Ammunition Plant, Lake City, Missouri, July 6, 1998 - July 17, 1998, submitted by Karen Myers, US Army Engineer Waterways Experiment Station, Vicksburg, MS

15. Thermal Desorption VOC Sampler Data Package for Longhorn Army Ammunition Plant, Karnak, Texas, 10 August 1998 - 21 August 1998, submitted by Karen Myers, US Army Engineer Waterways Experiment Station, Vicksburg, MS

16. Quality Assurance for Environmental Chemical Analyses, Environmental Chemistry Branch, Environmental Laboratory, U.S. Army Engineer Waterways Experiment Station, Vicksburg, MS, March 24, 1994.

17. ASTM Method D3441 -75 T, Tentative Method for Deep, Quasi-static, Cone and friction-Concentration Tests of Soil, approved August 29, 1975.

18. Method 5035, Closed-system Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, U.S. EPA SW-846 Third Edition, Revision 0, December 1996

19. Direct Sampling Ion-Trap Mass Spectrometry (DSITMS), Characterization, Monitoring and Sensor Technology Crosscutting Program, Innovative Technology Summary Report, Prepared for U.S. Department of Energy, Office of Environmental Management, December 1998.

20. Method 8260A Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Capillary Column Technique, US EPA SW-846 Third Edition, Revision 1, September 1994.

21. Draft Method 8265, Direct Sampling Ion-Trap Mass Spectrometry for the Measurement of Volatile Organic Compounds In Water, Soil, and Air, SW-846 Organic Methods Workgroup, Revision WG 2, July 1997.
22. Minutes from the July 7<sup>th</sup> 1997, SW-846 Organic Methods Workgroup, completed on November 19, 1997.
23. Draft SOP 732-S, Guide for Field Soil Sampling with Encore Sampler for VOC Analysis, California Department of Toxic Substances Control Hazardous Materials Laboratory, Revision No. 2, July 1998
24. New Jersey Department of Environmental Protection, Methodology for the Field Extraction/Preservation of Soil Samples with Methanol for Volatile Organic Compounds, December 1996.
25. U.S. Environmental Protection Agency, A Test Methods for Evaluating Solid Waste; Physical/Chemical Methods, SW-846 Third Edition, Washington, DC. 1984.
26. Multi-state Evaluation of the Site Characterization and Analysis Penetrometer System Volatile Organic Compound (SCAPS-VOC) Sensing Technologies, December 1997, Prepared by the Accelerated Site Characterization (ASC) Team, Subtask II, SCAPS New Sensor Development Review Team established under ITRC.
27. Comparison of Methods for Sampling Vadose Zone Soils for Determination of Trichloroethylene, Alan D. Hewitt, U.S. Army cold Regions Research and Engineering Laboratory, Journal of AOAC International Vol. 77, No. 2, 1994.
28. Field Detection & Process Monitoring, Teledyne Scientific Instrument, Mountain View, CA., undated.
29. Data Evaluation of Methanol Verification QC Samples Based on EPA Method 8260B (VOC by GCMS) from Lake City Army Ammunition Plant, Missouri, Longhorn Army Ammunition Plant, Texas, and US Army Corps Engineers (Project Name: SCAPS TDS-CCRL-QC), James Cheng, Hazardous Materials Laboratory, internal memo to Ruth Chang, November 19, 1999.
30. Tri-Service Site Characterization and Analysis Penetrometer System (SCAPS) Validation of the Thermal Desorption Sampler for Volatile Organic Compounds, Instruction Report, U.S. Army Engineer Research and Development Center, Environmental Laboratory. Vicksburg, MS. in press 1999 by Myers, K.F., Karn, R.A., Eng, D.Y., Konecny, K.F., Davis, W.M..



# **APPENDIX I**

## **SCAPS TDS FIELD STUDY RESULTS**

Hanover, Lake City, Longhorn and Davis Sites